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# FILTER PAPER STUDIES VI
## EFFECT OF VISCOSE AND ASBESTOS TYPES

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# FILTER PAPER STUDIES VI
## EFFECT OF VISCOSE AND ASBESTOS TYPES

Harold W. Knudson and Robert D. Parsons

Hollingsworth and Vose Company
East Walpole, Massachusetts

January 16, 1950

Approved by:

Dr. E. A. Ramskill, Head, Protective Chemistry Branch
Dr. P. Borgstrom, Superintendent, Chemistry Division

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# PREFACE

This Naval Research Laboratory report consists of the following two Research and Development Reports written by H. W. Knudson and R. D. Parsons of the Hollingsworth and Vose Company, East Walpole, Mass.,on Navy Contract N7-onr-430:

"Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy H-60 Filter Paper," Fifth Quarterly Period of Contract N7-onr-430, referred to as the N-11 Trial.

"Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy H-60 Filter Paper," Sixth Quarterly Period of Contract N7-onr-430, referred to as the N-12 Trial.

This report concludes the work on the first half of the extended contract. Filter paper studies are being continued by the Hollingsworth and Vose Company and additional reports will be published when received.

# CONTENTS

## ABSTRACT

This is an interim report describing in detail two mill runs and the associated laboratory research work on mill procedures for the manufacture of causticized viscose-asbestos filter papers. It is shown that bright viscose rayon may be used in place of the dull grade and that Blue African asbestos may be used in place of Blue Bolivian asbestos. The shrinkage problem on the machine driers is appreciably decreased by the use of bright viscose. The most efficient Navy filter paper ever made on a semi-production scale is described (N-11). The first trial of the Hydrapulper (N-12) is judged to be satisfactory.

## PROBLEM STATUS

This is an interim report; work on the problem continues.

## AUTHORIZATION

NRL Problem C04-28R (NS 181-011)

# RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

(N-11)

## INTRODUCTION

This report is a summary of the work done under contract N7-onr-430 from July 1, 1948 to September 30, 1948. A statement of the general objectives and an outline of previous work accomplished are contained in past reports covering the mill trials and laboratory work from April 1946 to July 1948.*

The mill trial reported here is designated as the N-11 trial, H & V Lot No. 694, dated November 15, 1948.

Reference to earlier reports will show that considerable progress has been made in the effort to develop a domestic substitute for esparto in the Navy Type H-60 filter material. For reasons set forth in previous reports recent attention has been centered on the use of causticized viscose rayon flock. Dull type viscose rayon (delustered) was used in most of this work, since the earlier

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*Knudson, H. W., "Filter Paper Studies I. Effect of Replacing Esparto with Yucca Fiber," NRL Report C-3172, September 1947. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies II. Effect of Replacing Esparto with Wood Pulp Fiber," NRL Report C-3225, January 1948. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies III. Effect of pH and Added Electrolytes," NRL Report C-3226, January 1948. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies IV. Effect of Replacing Esparto with Viscose Rayon Fiber," NRL Report C-3299, June 10, 1948. (Confidential)

Knudson, H. W., "Filter Paper Studies V. Effect of Viscose Fiber Processing," NRL Report C-3394, December 1948. (Confidential) studies had indicated that somewhat superior results were obtained with this type compared with the bright viscose.

However, subsequent work has demonstrated that some of the important variables which are now recognized were overlooked at that time. Although it had been planned to continue this work using the dull type for the present, the work was suddenly interrupted when it was learned that the DuPont Company intended to discontinue the manufacture of this grade. It thus became necessary to re-evaluate immediately the performance of the bright viscose in the gas mask filter material. The work described in this report deals chiefly with this problem.

It was also decided to work with Blue African asbestos instead of Blue Bolivian during this study. This decision was made for several reasons.

1. A recent survey of the supply situation indicates that the Blue African grade is more generally available and less subject to quality variation.

2. Less Blue African fiber is required than Blue Bolivian for the same performance in H-60 type paper.

3. Recently installed Vortrap units on the paper machine would permit a study of the dirt removed from Blue African asbestos. (It should be mentioned that the relatively large contamination of the African asbestos proved very troublesome in earlier work.)

4. The lesser amount of African asbestos required might lead to improved physical strength of any subsequent filter paper developed.

Pending the installation of the Navy Hydrapulper (Contract N8-onr-623), it was decided to continue to causticize the rayon in the beater rather than in the rotary boiler. In this connection, it can be reported that the construction of the Hydrapulper pit at the West Groton mill of Hollingsworth and Vose Company is under way and that present plans call for delivery and installation of the equipment by February, 1949. It is hoped that the Hydrapulper will permit much easier control of the causticizing process and represent a more practical approach to the production of commercial quantities of this new type of paper.

Not only does the causticized rayon promise to be an acceptable substitute for esparto in the Navy Type H-60 filter material, but it has already been demonstrated that the rayon type of papers are much more efficient from the standpoint of air resistance and smoke penetration. Thus it seems doubly important to concentrate on efforts to eliminate the many production problems associated with this type of paper.

## LABORATORY WORK

Since it became increasingly difficult to obtain dull viscose rayon fiber, it was necessary to study bright viscose as an alternate in the rayon furnish. Samples of bright cut viscose were procured and investigated for this type of filter paper. Almost all the difficulties encountered in causticizing and processing the dull viscose pertained to bright viscose rayon also. The most important of these processing variables are temperature control, caustic concentration, and the time and type of mechanical treatment.

It was thought that perhaps a 3-denier bright viscose fiber could be used in place of the 1.5-denier fiber. However, it was found that a higher caustic concentration was needed as well as a larger amount of asbestos. The strength of these sheets was very good but the filtration efficiencies were relatively low.

Waring Blendor Run

The 1.5-denier bright cut viscose was first studied through causticizings performed in the Waring Blendor to find out the caustic concentrations which would produce the best paper. The viscose was causticized in several caustic solutions having concentrations from 4.75% to 9.5%. Each causticizing was carried out in a Waring Blendor for 5 seconds, after which the viscose was quenched and thoroughly washed. Both 1/8″ cut and 1/4″ cut were used. However, the sheets containing one-half of each are reported here to show the average test values. A stock solution (0.5%) of Blue South African asbestos was prepared in the normal way. It should be stated, however, that it was not practical in the laboratory to efficiently clean the asbestos used. As a result, all the laboratory penetration efficiencies are probably on the conservative side, a point which will be checked in the mill trial.

Table I shows the smoke penetration, air resistance characteristics, and physical data for these handsheets. The viscose stock is made up of 50% 1.5-denier 1/8″ bright cut and 50% 1.5-denier 1/4″ bright cut as stated above.

TABLE I

Waring Blendor Run, Performance and Physical Characteristics vs. %NaOH

| Sample | % NaOH | % BSA | Resistance* (mm $H_2O$) | Penetration* (%) | Efficiency+ (%) | Caliper (in.) | Tensile (lb/in. width) |
|---|---|---|---|---|---|---|---|
| 1 | 4.75 | 14.0 | 72 | .092 | 4.22 | .043 | 1.5 |
| 2 | 6.0 | 15.0 | 85 | .016 | 4.48 | .037 | 1.6 |
| 3 | 6.5 | 15.0 | 103 | .010 | 3.88 | .040 | 1.8 |
| 4 | 7.0 | 15.0 | 124 | .004 | 3.55 | .038 | 1.9 |
| 5 | 7.5 | 15.0 | 147 | .001 | 3.40 | .030 | 2.3 |

*Measured by NRL Smoke Penetration Meter E2, E2R1, or E3. For operating instructions see NRL Instruction Manual A 825A, "Instructions for Canister Tests, Part II, Filters, Section A, Smoke Penetration," 13 July 1945.

+Percent efficiency = $\frac{-\log P}{R}$ x 100, where P is the DOP penetration expressed in decimals rather than percent, and R is the resistance across the sample in mm of water under the standard conditions of test.

It can be seen that the efficiency decreases as the caustic concentration increases. It will also be noted that the Waring Blendor gives a very vigorous type of agitation to these viscose fibers as compared with the action of a beater. The preliminary tests were primarily performed to find the range of caustic concentrations which would be most suitable. These concentrations would then be further studied in the laboratory beater.

From the data shown above it appears that there is a steady increase of strength as the caustic concentration increases.

Beater Run

Since it was known that the Blendor produces a more drastic action on the fibers than circulation in the beater, it was decided to begin beater causticizings at 6.5% caustic. A series of beater causticizings was made to find the optimum conditions which would produce a filter paper of the desired characteristics.

The furnish used in each run was composed of 75% 1.5-denier 1/8" bright cut and 25% 1.5-denier 1/4" bright cut viscose. This stock was circulated with the roll up and a 4% consistency in the beater for 10 minutes. A temperature of 75°F was selected for all runs because it is believed that it can be realized in production without much difficulty in summer as well as winter. Working at one temperature eliminates an important variable, which heretofore has caused much apprehension in the causticizing operation.

It is now believed that if the stock were circulated only until the fibers were separated and wetted out, the resultant sheets would have had higher efficiencies. Further laboratory investigation is needed to minimize the time of causticizing for any given caustic concentration.

The specific gravities of each caustic solution used in this study were carefully recorded at the operating temperatures. The water temperature at this time of year is well below 60°F so that no trouble was anticipated in causticizing at 75°F.

In Table II are reported the penetration, air resistance, and physical data for the laboratory beater runs.

TABLE II

Beater Run, Performance and Physical Characteristics vs. %NaOH

| Sample | % NaOH | % BSA | Air Resistance (mm $H_2O$) | Average Penetration (%) | Average Efficiency (%) | Average Caliper (in.) | Average Tensile (lb/in. width) |
|---|---|---|---|---|---|---|---|
| 1 | 6.5 | 17.0 | 95 | .025 | 3.79 | .040 | 2.0 |
| 2 | 6.8 | 16.0 | 84 | .086 | 3.74 | .034 | 1.7 |
| 3 | 7.0 | 18.0 | 89 | .035 | 3.87 | .041 | 2.0 |
| 4 | 7.10 | 15.0 | 103 | .020 | 3.71 | .030 | 2.5 |
| 5 | 7.25 | 15.0 | 87 | .049 | 3.85 | .033 | 2.9 |
| 6 | 7.50 | 16.0 | 93 | .028 | 3.83 | .037 | 3.0 |

It appears from the above data that a concentration of 7.25 to 7.5% caustic produced the best sheet with a minimum amount of asbestos. Higher caustic concentrations led to gel formation and difficult washing. It should also be noted that the retention of the asbestos is about 8% lower than the recorded percentage added. This is true for all sheets of this type made in the laboratory. Several ash determinations were made to substantiate this point.

It can be seen that here again the last two samples possess greater tensiles, and are therefore more satisfactory. It was also observed that these sheets made from bright rayon possessed fair scuff resistance and excellent folding properties.

Throughout the present investigation it was noted that the bright rayon handled quite differently from the dull rayon. Even the general appearance during processing was quite different. Perhaps the greatest difference to be noted relates to the shrinkage which occurs when the handsheets were dried. In this respect, sheets made from causticized bright rayon showed considerably less shrinkage than those made from the dull. This observation was especially encouraging in view of the very great trouble experienced in earlier mill trials due to the excessive shrinkage over the dryer section of the paper machine. (It will be remembered that causticized rope fibers were used in the N-10 trial to minimize this shrinkage.) Further study of this characteristic of bright rayon in the laboratory led to the conclusion that it might be possible to compensate the paper machine for the lesser amount of shrinkage without resorting to the use of "diluting" fibers such as causticized rope.

## THE MILL RUN

### Furnish

With this information, it was decided to make a mill run using the following base furnish:

100% Causticized Rayon Flock
75 parts 1.5-denier 1/8" bright
25 parts 1.5-denier 1/4" bright

To this would be added approximately 10% Blue South African asbestos on the weight of the base furnish. A quantity of causticized rope was to be kept on hand in case of excessive shrinkage on the machine.

### Preparation of Stock for Mill Trial

On the basis of laboratory studies, it was decided to causticize two beaters of bright viscose, 500 pounds each, at a caustic concentration of 7.25% and 7.50% respectively. A quantity of water just under the total amount needed was added to the beater, and a quantity of 50% caustic solution was added. The makeup water was then added to bring down the concentration to 7.25%. At this point the temperature was approximately 67°F. A steam line was put into the beater to bring the temperature up to 75°F. When the temperature and concentration had been adjusted, 500 pounds of bright viscose (75 parts 1.5-denier 1/8", 25 parts 1.5-denier 1/4") was added to the beater. The beater roll was kept raised throughout the operation. The consistency was about 4%. The stock was circulated in the beater until the fiber bundles were separated and entirely wetted out. At the end of approximately 8 minutes the causticizing was judged to be complete, and the beater was dumped into a chest containing 3 times the beater volume of water. After this quenching the stock was pumped over the Fourdrinier section of the paper machine and removed as wet lap. This wet lap was transferred back to the beater for a final washing. After an hour of washing the viscose stock registered a pH of 6.8. At this point the stock was considered washed, and it was dropped to the beater chest.

The beater was then furnished with 25 pounds of Blue South African asbestos and beaten hard for 1 hour until it was well defibered. This asbestos was then dropped into the beater chest and mixed with the viscose furnish before going over the machine. Another 15 pounds of asbestos was prepared the same way and pumped to the auxiliary chest for bleeding in at the headbox as required.

After the processing of this first beater, another quantity of viscose was causticized in like manner at a 7.50% caustic concentration and 75°F. This stock was made into wet lap and washed in the beater to a pH of 7.0. 28 pounds of Blue South African asbestos was then beaten hard for an hour and dropped to the beater chest containing the above viscose.

The average yield of the two beaters of causticized viscose was approximately 76%. Handsheets were prepared from the two plain viscose stocks after being washed. The physical characteristics of these sheets were very similar.

Manufacturing Data

The constituents of each of the two beaters were kept separate in the beater chests in spite of the fact that preliminary handsheets indicated very little difference between them. Each beater contained the following furnish:

| Beater No. 1 | | Beater No. 2 | |
|---|---|---|---|
| 360 lb | causticized and washed bright viscose rayon (75% 1/8″ cut, 25% 1/4″ cut, 1.5-denier) | 400 lb | causticized and washed bright viscose rayon (75% 1/8″ cut, 25% 1/4″ cut, 1.5-denier) |
| 25 lb | beaten Blue South African asbestos | 28 lb | beaten Blue South African asbestos |

The stock went through a Jordan under pressure with the plug backed off to insure thorough mixing and was sent through the Vortraps for the additional removal of dirt from the asbestos. Approximately 4% additional asbestos was added to the stock from the auxiliary asbestos tank. This asbestos was fed sparingly at the beginning of the run.

One roll on the calender stack was used to soften up the sheet. No difficulty was experienced in the formation and handling of this paper once the machine was set up. Most of the first beater was over the machine by the time the Vortraps, asbestos, ream weight, and other machine variables had been adjusted satisfactorily.

Performance of the Paper

The physical properties of this paper are as follows:

a) Caliper - 0.043 in.
b) Ream Weight - 159.9 lb
c) Length Tensile - 1.0 lb
d) Moisture - 5.0%

These are average values. The tensile is somewhat low, but no great accuracy can be expected in such a range on the standard testers employed.

TABLE III

Smoke Filtration Characteristics of Paper from Mill Run

| Sample | Resistance (mm $H_2O$) | Penetration (%) | Efficiency (%) | Remarks |
|---|---|---|---|---|
| 1 | 68 | .140 | 4.22 | Adding more auxiliary asbestos |
| 2 | 108 | .008 | 3.80 | Too much moisture in paper |
| 3 | 113 | .001 | 4.43 | Second beater starting— driers functioning properly |
| 4 | 102 | .003 | 4.44 | Cutting back asbestos— Vortraps working properly |
| 5 | 93 | .008 | 4.41 | |
| 6 | 81 | .021 | 4.55 | Less asbestos |

Since there was no excessive shrinkage during the trial, no causticized rope was added. It can be seen from Table III that causticized bright viscose alone with South African asbestos produced a filter paper with a higher efficiency than in any previous Navy mill trial. These efficiencies are somewhat higher than those recorded from laboratory handsheets.

TABLE IV

Effect of DOP Exposure on Performance, N-11 Trial Sample

| Time (min) | Resistance (mm $H_2O$) | Penetration (%) |
|---|---|---|
| 0 | 92.0 | .005 |
| 2 | 94.5 | .004 |
| 4 | 95.5 | .004 |
| 6 | 96.0 | .004 |
| 8 | 96.5 | .004 |
| 10 | 97.0 | .004 |

The rate of "break" was exceptionally low and is regarded as being entirely satisfactory.

TABLE V

Performance vs. Flow Rate, N-11 Trial Sample

| Sample Flow Rate (l/min) | Resistance (mm $H_2O$) | Penetration (%) |
|---|---|---|
| 85 | 99 | .004 |
| 42-1/2 | 49-1/2 | .003 |
| 21-1/4 | 25 | .002 |

The above data show a normal decrease in penetration with flow rate, which is an indication of satisfactory asbestos distribution and absence of pin holes in this paper.

Effect of Aging

Samples from the N-11 mill trial were retested two weeks after manufacture to find out whether any relaxation occurred on storage.

TABLE VI

Effect of Aging on Performance, N-11 Trial Samples

| Interval After Manufacture | Resistance (mm $H_2O$) | Penetration (%) |
|---|---|---|
| None (sample 1) | 100 | .003 |
| 14 days (sample 1) | 105 | .003 |
| None (sample 2) | 95 | .005 |
| 14 days (sample 2) | 92 | .009 |

Table VI indicates little if any relaxation. It is perhaps unfortunate that more samples were not saved for this series of studies. Several samples from one of the parent rolls were tested recently without the benefit of having tests on sister sheets at the time of manufacture. This information led to the observation that the paper did not test uniformly over different areas of the same sample, especially when using the small test fixture on the E-3 Smoke Penetration Meter. Although this phenomenon has not been observed before, it is believed that it represents nothing over which to express alarm. The variations in smoke penetration were of the order of .005-.008% at absolute penetrations of the same order. Whereas the percentage variations were extremely high, it must be remembered that similar variations in the standard H-60 paper (.050 - .075% penetration) would almost go unnoticed. It should be pointed out further that the absolute penetrations of the N-11 samples were in the range where the accuracy of the present smoke penetration meter and particle size control are open to some question.

If any relaxation does occur it is certainly of small order. Actually, present evidence indicates that any changes in smoke penetration and resistance which occur on standing are of the same order as the natural occurring small variations in uniformity associated with commercial production of paper. An effort will be made to investigate this problem further during the next mill trial.

## DISCUSSION

From the present laboratory and mill experience, it is believed that the bright viscose rayon can be used as successfully as the dull grade. In the case of the bright, the shrinkage effect is somewhat less and permits easier adjustment of the differential speeds of the cans in the dryer section of the paper machine. It is believed the tensile strength of sheets made from the bright are somewhat inferior to those made from the dull rayon. However, the difference is small between the machine made papers and difficult to evaluate properly. Actually, the physical strength of the N-11 paper was adequate to handle it over the paper machine and rewinders. This has sometimes been used as a criterion for adequate strength.

Should this new type of filter material represent an item on which the Navy would ultimately standardize, steps should be taken to survey the manufacturing capacity for this particular grade of bright viscose rayon. It would be unfortunate to have a situation develop similar to the one reported here where the dull viscose rayon was discontinued as a standard item. Representatives of the flock cutting industry (not rayon manufacturers) have told us that the 1.5-denier bright rayon represents a standard grade available in large amounts.

The unusually high efficiency of the N-11 paper is not attributed directly to the use of bright rayon. The added efficiency is thought to have been gained through the use of the Vortrap-cleaned Blue South African asbestos. There can be no doubt that the Vortraps performed an outstanding job in removing foreign material from the asbestos. Periodic examination of the rejected material revealed relatively large quantities of gravel-like material and unopened fibers. Through the use of African asbestos less total asbestos fiber was required. This should have the effect of adding strength to any given paper over using Blue Bolivian asbestos. Other advantages of Blue African asbestos have been listed in an earlier section.

In general the N-11 paper is considered quite outstanding from the standpoint of filtration efficiency, resistance to break, decreased penetration with flow rate, and folding characteristics. Improved mechanical strength, scuff resistance, and uniformity would be desirable.

# RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

(N-12)

## INTRODUCTION

This report is a summary of the laboratory work done under contract N7-onr-430 from October 1, 1948 to December 31, 1948 and the N-12 mill trial. A statement of the general objectives and an outline of previous work accomplished are contained in past reports covering mill trials and laboratory work from April 1946 to September 1948.*

The mill trial reported here is designated as the N-12 trial, H & V Lot No. 859, dated March 25, 1949. This mill trial, N-12, was necessarily delayed pending the installation of the Navy Hydrapulper (Contract No. N8-onr-623). As stated previously, the plans had called for delivery and installation of this equipment by February, 1949. However, installation was not completed until the middle of March, 1949.

Reference to earlier reports will show that considerable progress has been made in the effort to develop a domestic substitute for esparto in the Navy Type H-60 filter material. For reasons set forth in previous reports, recent attention has been centered on the use of causticized viscose rayon flock. Dull type viscose rayon (delustered) was used in most of this work, since earlier studies had indicated that somewhat superior results were obtained with this type compared with the bright viscose. However, for reasons set forth in the last report bright viscose rayon flock was thoroughly investigated. Since the supply of bright viscose seemed to be adequate and also because its performance in viscose type paper met with success, it was decided to incorporate bright viscose rayon in the remaining mill trials of this contract No. N7-onr-430. Since it has been demonstrated in previous mill trials that the rayon type of filter papers are the most efficient, it was decided that future work was to be concentrated on the production problems associated with this type of paper. A major step in this direction seemed to be the installation of a Hydrapulper which would be used in large measure to help standardize the causticizing procedure.

The primary objective of this and future mill trials now seems to be the attainment of knowledge and experience in the operation of the new Hydrapulper, in the production of viscose-type filter material.

The use of Blue African asbestos was continued for the reasons outlined in the N-11 report, since the Vortrap units did an outstanding job in removing foreign material from the asbestos.

*NRL Reports C-3172, C-3225, C-3226, C-3299, C-3394. (all Confidential)

In connection with production of filter paper, there has been some laboratory evidence that washed causticized viscose stock may lose tensile strength upon standing more than 24 hours. This observation would suggest that the viscose rayon be causticized and processed as rapidly as possible. It is believed that the Hydrapulper, used in place of the beater or a rotary boiler, would expedite these operations to a great extent.

## LABORATORY WORK

Since the use of the Hydrapulper was anticipated for this mill trial, work in the laboratory was centered around its operation for the causticizing of bright cut viscose rayon.

The Waring Blendor was used in the laboratory to simulate the mechanical action of the Hydrapulper and thus establish the causticizing conditions to be used in the latter. It had been decided previously that the optimum causticizing temperature would be 75°F, taking into account the seasonal variation in temperature of the process water. From past experience with the Waring Blendor it was thought that a caustic concentration of 7.5% at 75°F would be an optimum concentration to evaluate a variation in the time of causticizing.

In all causticizings performed in the laboratory a 1.5-denier bright cut viscose rayon was used (75% of 1/8" fibers and 25% of 1/4" fibers). The asbestos was beaten and defibered in a 2% (on fiber weight) solution of Daxad No. 11, a dispersing agent. It has been found that this dispersing agent incorporated in the asbestos tends to improve the filtration performance of the rayon-asbestos sheet. This result is thought to be due primarily to a slightly better dispersion of asbestos in the paper. In any event, the addition of this dispersing agent does not seem to have any detrimental effect on the furnish or the finished sheet of paper.

Table I shows the smoke penetration, air resistance and physical characteristics of handsheets made from bright viscose rayon causticized at 75°F in 7.5% sodium hydroxide in the Blendor for a varying length of time. The consistency during causticizing was 4%.

TABLE I

Performance and Physical Characteristics vs. Time of Causticizing

| Sample | Time of Causticizing (sec) | % BSA | Resistance (mm $H_2O$) | DOP Penetration (%) | Efficiency (%) | Caliper (in.) | Tensile (lb./in. width) |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 9.0 | 78 | .059 | 4.16 | .045 | 1.6 |
| 2 | 10 | 9.0 | 95 | .025 | 3.82 | .037 | 1.6 |
| 3 | 15 | 9.0 | 104 | .014 | 3.72 | .037 | 1.9 |
| 4 | 60 | 8.0 | 104 | .012 | 3.83 | .037 | 4.9 |
| 5 | 120 | 8.0 | 96 | .028 | 3.71 | .040 | 7.8 |

It can be seen that the efficiency decreases slightly as the time of causticizing increases. It will also be noted that the amount of asbestos needed decreases slightly with increased time. These tests were performed mainly to find out what effect time had on the efficiency and strength of the resultant sheets.

From the data given above it appears that there is an appreciable increase of strength when the time of causticizing is increased.

Because the efficiency was gradually lowered as the tensile strength was raised, an optimum causticizing condition had to be decided upon to give the most desirable filter properties. This in itself was rather a problem since it was known that the types of mechanical agitation of the Blendor and Hydrapulper are not identical. The Blendor impeller attains a much higher velocity than that developed in the Hydrapulper. Also the ratio of the areas between the containers and impellers are considerably different. Consequently it was believed that longer causticizing was needed in the Hydrapulper than in the Blendor, other conditions being constant.

In order to determine what effect the speed of the Blendor impeller had in the causticizing of viscose rayon, a Variac (Type V-5MT) was procured and put in series with the Blendor.

In Table II are recorded the air resistance, penetration, and physical data for handsheets made from viscose causticized under the same conditions mentioned in the last experiment except that time was kept constant at 10 seconds, and only the speed of the impeller, reported as output voltage of the Variac, was varied.

TABLE II

Performance and Physical Characteristics vs. Impeller Speed

| Sample | Variac Reading (Output Voltage) | % BSA | Air Resistance (mm $H_2O$) | Penetration (%) | Efficiency (%) | Caliper (in.) | Tensile (lb/in. width) |
|---|---|---|---|---|---|---|---|
| 1 | 80 | 9.0 | 95 | .025 | 3.82 | .037 | 1.6 |
| 2 | 90 | 9.0 | 134 | .0008 | 3.81 | .042 | 3.8 |
| 3 | 100 | 8.0 | 101 | .0075 | 4.08 | .039 | 4.0 |
| 4 | 110 | 8.0 | 103 | .007 | 4.02 | .040 | 4.2 |

It appears from this that change in Blendor speed produced little change in efficiency. Subsequent tests showed, however, that there is an appreciable drop in efficiency with increasing time of causticizing.

It can be seen here that the tensile strength increases with higher impeller velocities. There is little doubt that these greater strengths are obtained solely by this type of mechanical agitation. Moreover, there seems to be a minimum amount of gel formed in these time and velocity ranges.

## THE MILL RUN

Using the information obtained in the laboratory and from mill trial N-11 concerning the behavior of bright viscose rayon it was decided to make the following mill run using the Navy Hydrapulper.

### Furnish

The base furnish was to consist of 100% causticized Rayon Flock (75 parts 1.5-denier 1/8" bright, 25 parts 1.5-denier 1/4" bright). To this would be added approximately 10% Blue South

25 lb Blue South African asbestos beaten with 4% Daxad No. 11

The stock went through a Jordan under pressure with the plug backed off to insure thorough mixing and was sent through the Vortraps for the additional removal of dirt from the asbestos. Between 4 to 6% additional asbestos was added to the stock from the auxiliary asbestos tank. Very little asbestos was fed in at the beginning of the run. The white water return was used as much as possible.

The last section of driers on the paper machine was lagged in order to prevent excessive tension where the paper exhibited the most shrinkage. One roll on the calender stack was used to soften up the sheet. Little difficulty was experienced in the formation and handling of this paper after the machine was once set up. Most of the first chest was over the machine by the time the asbestos content, ream weight, and other machine variables had been adjusted satisfactorily.

Performance of the Paper

Average values of the physical properties of this paper are as follows:

a) Caliper - 0.038 in.
b) Ream Weight - 134 lb
c) Length Tensile - 2.3 lb
d) Moisture - 5.0%

The tensile strength was very satisfactory.

TABLE III

Smoke Filtration Characteristics of Paper from Mill Run

| Sample | Resistance (mm $H_2O$) | Penetration (%) | Efficiency (%) | Remarks |
|---|---|---|---|---|
| 1 | 88 | .040 | 3.87 | Low weight |
| 2 | 85 | .035 | 4.06 | Slightly damp |
| 3 | 96 | .017 | 3.92 | Dry paper |
| 4 | 108 | .006 | 3.91 | 150 lb ream weight |
| 5 | 115 | .005 | 3.74 | ---------------- |
| 6 | 140 | .004 | 3.36 | Heavy paper |
| 7 | 121 | .002 | 3.88 | Losing weight |
| 8 | 97 | .009 | 4.18 | Some foam spots |

Since there was no excessive shrinkage during the trial, no causticized rope was added. It can be seen from Table III that bright viscose causticized in the Hydrapulper together with South African asbestos produced a filter paper with very satisfactory filtration characteristics. These efficiencies are quite comparable with those recorded from laboratory handsheets.

TABLE IV

Effect of DOP Exposure on Performance, N-12 Trial Sample

| Time (min) | Resistance (mm $H_2O$) | Penetration (%) |
|---|---|---|
| 0 | 105 | .008 |
| 2 | 108 | .009 |
| 4 | 108 | .010 |
| 6 | 109 | .010 |
| 8 | 111 | .010 |
| 10 | 111 | .010 |

The rate of "break" was considered normal and is regarded as entirely satisfactory.

TABLE V

Performance vs. Flow Rate, N-12 Trial Sample

| Sample Flow Rate (l/min) | Resistance (mm $H_2O$) | Penetration (%) |
|---|---|---|
| 85 | 103 | .008 |
| 42-1/2 | 51-1/2 | .008 |
| 21-1/4 | 25-1/2 | .008 |

Although the percent penetration should have decreased with decreasing flow rate, these data show the penetration to remain the same. If these data are correct, they would indicate a non-uniform distribution of asbestos in the paper which in extreme cases leads to an increase in penetration with decreasing flow rate. This effect has sometimes been referred to as the "pin-hole" effect. The distribution of asbestos in the N-12 paper is not as good as we have realized in some of the earlier papers and this probably accounts for the observed data.

There are adjustments which can be made on the Fourdrinier wire during the felting of the paper which usually lead to an improved asbestos distribution. Unfortunately it was not possible to make these adjustments during this trial for reasons discussed in the last section of this report. It should be stated, however, that this difficulty was the result of an oversight in diluting the stock to a consistency below normal. The fact that Daxad No. 11 was used to aid the dispersion of asbestos during the beating cycle may have aggravated the situation, but no similar effect has been noted in the laboratory.

Effect of Aging

The samples were retested four days after manufacture, an interval which should be sufficient to point out any relaxation that has occurred in the paper during storage. Table VI shows the air resistance and smoke penetration of the N-12 paper at the time of manufacture and four days later. It also shows the relaxation, if any, between the third and fourth day after manufacture.

TABLE VI

Effect of Aging on Performance, N-12 Trial Samples

| | Date of Manufacture | | Three days later | | Four days later | |
|---|---|---|---|---|---|---|
| Sample | Resistance (mm $H_2O$) | Penetration (%) | Resistance (mm $H_2O$) | Penetration (%) | Resistance (mm $H_2O$) | Penetration (%) |
| 1 | 115 | .005 | | | 106 | .007 |
| 2 | 105 | .012 | | | 98 | .016 |
| 3 | 106 | .010 | | | 101 | .012 |
| 4 | 110 | .005 | | | 102 | .009 |
| 5 | | | 98 | .006 | 100 | .005 |
| 6 | | | 102 | .004 | 100 | .005 |
| 7 | | | 101 | .005 | 104 | .004 |
| 8 | | | 88 | .011 | 90 | .010 |

Table VI indicates there is little relaxation and that any relaxation that did occur must have done so within the three days after manufacture. Although the first four samples show an appreciable decrease in resistance after four days, the filtration efficiencies of the paper are actually increased a little.

## DISCUSSION

It is believed that this mill trial carried out with the use of the Navy Hydrapulper was highly successful. The primary purpose of this trial was to gain experience in the operation of the Hydrapulper during the causticizing procedure. The knowledge obtained from using the pulper would be extremely valuable in correlating work done with the Waring Blendor in the future.

During the causticizing operation samples were taken at 1, 2, and 5 minutes of agitation in the Hydrapulper. Upon examination of handsheets made from these samples it could be seen that there were unopened viscose bundles present in the sheets made after 1 minute agitation. Sheets made from the sample after 2 minutes did not contain these unopened bundles of fiber. The 5 minute samples exhibited the highest tensile strength. It is now believed that causticizings up to 10 minutes or more would be entirely satisfactory.

Inadvertently the stock was diluted to such an extent in the chests that by the time it ran over the wire the consistency was too low to permit the optimum circulation of white water. The use of a somewhat larger quantity of asbestos in this trial is probably due to the higher loss of asbestos through the screen. Had it been possible to re-use more of the white water, this loss would have been minimized. It is believed that this loss can be reduced in future trials by maintaining a higher consistency in the machine chest. This higher dilution on the wire probably had a detrimental effect on the proper dispersion of asbestos throughout the sheet, since there was less opportunity to adjust the drainage on the wire. Also because of the dilution the machine was harder to set up to the proper weight of paper. A large percentage of paper made from the first batch of viscose, as well as being underweight, was slightly damp since the drier temperatures were raised gradually. This damp paper was, of course, harder to handle through the stack and rewinders.

The Hydrapulper stock washed very well in the beater. There did not seem to be any formation of an appreciable amount of gel in this process.

In general the N-12 trial is believed to represent a distinct improvement for the production of this filter paper on a commercial scale. It has a very satisfactory filtration efficiency, resistance to break, and folding characteristics. The mechanical strength and scuff resistance are greatly improved over N-11 paper. This N-12 paper has uniformity as good as any previous viscose type paper, but more improvement along this line is desirable.

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Filter paper studies VI. Effect of viscose and asbestos types, by Harold W. Knudson and Robert D. Parsons. Washington, Naval Research Laboratory, January 16, 1950.

17 pp. illus. 27 cm.

Abstract: Two mill runs and the associated laboratory research work are described on mill procedures for the manufacture of causticized viscose-asbestos papers. It is shown that bright viscose rayon may be used in place of the dull grade and that Blue African asbestos may be used in place of Blue Bolivian. The shrinkage problem on the machine driers is appreciably decreased by the use of bright viscose. The most efficient Navy filter paper ever made on a semi-production scale is described (N-11). The first trial of the Hydrapulper (N-12) is judged to be satisfactory.

1. Paper - filter. 2. Paper - manufacture. I. Hollingsworth and Vose Co. II. Contract N7-onr-430. III. Knudson, H. W. IV. Parsons, R. D. V. Filter paper studies.

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ABSTRACT:

Progress made in the development of a domestic substitute for esparto fiber in the Navy type H-60 filter paper is outlined. Two mill runs and the associated laboratory research work are described on mill procedures for the manufacture of causticized viscose-asbestos filter papers. It is shown that bright viscose rayon may be used in place of the dull grade and that Blue African asbestos may be used in place of Blue Bolivian. The shrinkage problem on the machine driers is appreciably decreased by the use of bright viscose. The most efficient Navy filter paper made on a semiproduction scale is discussed. Operation of the Hydrapulper, used in the pro[illegible]tion of viscose-type filter material, was satisfactory.


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